

JIS

JAPANESE INDUSTRIAL STANDARD

Identification of Dyestuff Classes on Dyed Textiles

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Identification of Dyestuff Classes on Dyed Textiles L 1065-1983

1. Scope

This Japanese Industrial Standard specifies the identification methods for the class of dye on dyed textiles.

2. Principle

Generally this standard applies to the dyed textiles from which the test specimen of a single nature of fibre having been dyed with a single class of dye can be taken out. This standard, however, gives, in some cases, facilities for the anticipation, to some extent, several classes of dye used on a test specimen.

This identification is done by the following procedure: Identify the nature of the fibre constituting the test specimen by the method specified in JIS L 1030 and then extract the dye from the test specimen or decolorize the test specimen by the use of the identification methods which have been laid down according to the nature of fibre. By the aids of the results thus obtained, such as change in colour or extraction degree of dye, or by the dyeing method using the extract liquid, identify the dye class used for the test specimen.

3. Devices and Materials

The devices and materials to be used for the methods shall be as specified below:

- (1) Test tube As specified in JIS R 3503.
- (2) Bunsen burner
- (3) Filter paper As specified in JIS P 3801.
- (4) Beaker As specified in JIS R 3503.
- (5) Pipette
- (6) Lead acetate paper
- (7) Watch glass
- (8) Evaporating dish As specified in JIS R 1302.
- (9) Porcelain crucible As specified in JIS R 1301.
- (10) Microscope
- (11) Separating funnel As specified in JIS R 3503.
- (12) Water bath
- (13) Flask with reflux condenser As specified in JIS R 3503.

Applicable Standards: See pages 32 and 33.

- (14) Undyed cotton cloth As specified in JIS L 0803.
- (15) Undyed wool cloth As specified in JIS L 0803.
- (16) Undyed acetate cloth As specified in JIS L 0803.

4. Reagents

Use the first or special grade reagent, if specified in JIS Standard.

- (1) Aqueous ammonia As specified in JIS K 8085.
- (2) Sodium chloride As specified in JIS K 8150.
- (3) Hydrochloric acid As specified in JIS K 8180.
- (4) Sodium hydroxide As specified in JIS K 8576.
- (5) Sodium hydrosulfite (hydrosulfite) As specified in JIS K 8737.
- (6) N, N-dimethylformamide As specified in JIS K 8500.
- (7) Pyridine As specified in JIS K 8777.
- (8) Sodium carbonate, 10 hydrate As specified in JIS K 8624.
- (9) Sodium nitrate As specified in JIS K 8562.
- (10) Nitric acid As specified in JIS K 8541.
- (11) Stearic acid As specified in JIS K 8585.
- (12) Urea As specified in JIS K 8731.
- (13) Acetic acid (99 to 100 %) (glacial acetic acid) As specified in JIS K 8355.
- (14) Diethyl ether As specified in JIS K 8103.
- (15) Tannic acid As specified in JIS K 8629.
- (16) Antimonyl potassium tartrate (tartar emetic) As specified in JIS K 8533.
- (17) Sodium sulfide As specified in JIS K 8949.
- (18) Lead acetate As specified in JIS K 8374.
- (19) Glycerol As specified in JIS K 8295.
- (20) Stannous chloride As specified in JIS K 8136.
- (21) Sodium azide As specified in JIS K 9501.
- (22) Iodine As specified in JIS K 8920.
- (23) Sodium hypochlorite

- (24) Zinc powder As specified in JIS K 8013.
- (25) Ethanol (99.5) As specified in JIS K 8101.
- (26) Sodium nitrite As specified in JIS K 8019.
- (27) β -naphthol (2-naphthol) As specified in JIS K 8699.
- (28) N-methyl-2-pirolidone
- (29) Ethyl salicylate
- (30) Chromium fluoride
- (31) Toluene As specified in JIS K 8680.
- (32) Nonionic dispersant
- (33) Tetrahydrofuran
- (34) Ligroin As specified in JIS K 8937.
- (35) Formic acid As specified in JIS K 8264.
- (36) Resolcinol As specified in JIS K 9032.
- (37) Chloroform As specified in JIS K 8322.
- (38) Dioxane (diethylene dioxide) As specified in JIS K 8461.

5. Preparation of Test Specimens

5.1 When preparing the test specimens, take them from a portion of the sample where it is presumed that a single class of dye is used, as far as possible.

If a sample is of different colour tones, prepare the individual test specimens containing every colour tone.

5.2 If the sample is a mixed textile, identify the individual fibres constituting the textile perfectly to classify them into every same nature of fibre, and prepare the individual test specimens of same fibre, as a rule.

5.3 Generally the test specimens shall weigh 0.05 to 0.30 g mass, and the undyed yarn or cloth shall weigh 0.01 to 0.05 g mass, as a rule.

5.4 If it is clear that the sample has been resin finished or treated after resin finishing, the treatment described below shall be made.

- (1) For Formaldehyde-amino Resin After treating the sample with 0.25 % solution of hydrochloric acid of 100 times the volume as that of the sample at 70 to 80°C for 15 min, take it out, wash it with warm water, 0.2 % aqueous ammonia, and water in this order, and after drying, make the test specimen.

- (2) For Resin of Acryl, Polystyrene and Metacryl Boil the sample with dioxane of 50 to 100 times volume as that of the sample in a flask with reflux condenser for 1 h, and after taking it out, wash and dry.
- (3) When Treatments Both with Dioxane and with Hydrochloric Acid Are Necessary Dioxane treatment shall be made first⁽¹⁾.

Note ⁽¹⁾ By this treatment, naphthol dye and some kinds of vat dye are extracted.

5.5 For some samples which have been subjected to other treatment such as waterproofing and may thereby bring forth any trouble on the dye classification, the test specimen shall be made after subjecting the sample to suitable treatment to facilitate the classification.

6. Dye Classes Capable of Being Classified According to Nature of Fibres Constituting Dyed Textiles

This standard specifies the identification methods for dye classes, which can classify the dye according to the nature of fibres, as enumerated in Table 1.

Table 1

Nature of fibre Dye class	Cellu- lose ⁽²⁾	Silk	Wool	Acet- ate	Nylon	Viny- lon	Acrylic and modacrylic	Poly- ester	Vinyl- dene	Polyvinyl chloride	Promix	Poly- chial
Direct dye	7.1.2 (1.2)	7.2.3	7.3.3		7.5.6	7.6.1						
Direct dye(metal after- treatment or metalline)	7.1.2 (1.2)											
Direct dye (diazotation developing)	7.1.2 (1.2)											
Acid dye		7.2.4	7.3.4		7.5.8		7.7.5				7.11.2	
Acid mordant dye		7.2.6	7.3.6		7.5.9		7.7.6					
Mordant dye		7.2.10	7.3.6									
1:1 type metal complex acid dye		7.2.7	7.3.7		7.5.5							
1:2 type metal complex acid dye		7.2.7	7.3.7		7.5.5	7.6.7	7.7.3				7.11.3	
Basic dye	7.1.3 (1.3)	7.2.5	7.3.5		7.5.7		7.7.4	7.8.2			7.11.4	
Sulfur dye	7.1.4 (1.4)					7.6.4						
Vat dye	7.1.5 (1.5)	7.2.9	7.3.9		7.5.2	7.6.5						
Solubilized vat dye	7.1.5 (1.5)	7.2.9	7.3.9		7.5.2							
Sulfur vat dye	7.1.4 (1.4)					7.6.4						
Naphthol dye	7.1.8 (1.6)			7.4.5	7.5.4	7.6.6						
Disperse dye				7.4.2	7.5.3	7.6.2	7.7.2	7.8.3	7.9.1	7.10.1	7.11.5	7.12.1
Developing disperse dye				7.4.3		7.6.3		7.8.3				
Reactive dye	7.1.9 (1.8)	7.2.8	7.3.8									
Oxidation dye(such as aniline black, etc.)	7.1.6											
Mineral dye(khaki coloured)	7.1.7											
Pigment resin colour	7.1.10 (1.7)			7.4.4		7.6.8	7.7.7	7.8.4	7.9.2	7.10.2	7.11.6	7.12.2

Note (2) This term means cotton, flax, hemp, ramie, jute, etc., viscose rayon and cuprammonium rayon generically.

Remark: There are specified the identification methods of dye class at the clauses of this standard numbered as in the above table. The number given in parentheses in the table indicates the clauses numbered in Appendix to the end of this standard.

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7. Identification Method

7.1 For Cellulose Fibre

7.1.1 Sequence of Identification Method Make the tests in the sequence given in Table 2, or make the tests in accordance with Appendix.

Table 2

Boil test specimen in aqueous ammonia which have been obtained by adding 0.5 to 1.0 ml of concentrated aqueous ammonia to 5 to 10 ml of water.

When dye is extracted	When dye is extracted only a little or not at all, make the test below for a fresh test specimen.		
	When dye is extracted	When dye is extracted only a little or not at all, make the test below for a fresh test specimen.	When any change can scarcely be found in test specimen
Make the dyeing test for extract liquid following 7.1.2 (direct dye).	Make the re-coloring test by using extract liquid of test specimen in boiling glacial acetic acid and then the dyeing test following 7.1.3(basic dye).	After boiling the test specimen in 1 % chloric acid solution, boil the specimen in 1 % aqueous ammonia solution.	Make the coloration test using concentrated nitric acid drops, following 7.1.5(2) (indanthrene blue). Make the ash test and mineral acid boiling following 7.1.7(mineral dye). Make the pyridine boiling and fluorescent test following 7.1.8(a part of naphthol dye), following 7.1.9 (a part of reactive dye) and following 7.1.10 (pigment resin colour).
	When dye is extracted somewhat or remarkably	When dye is extracted only a little or not at all, make the test below for a fresh test specimen.	
	Make the dyeing test for the extract liquid following 7.1.2 (apart of direct dye). Make the treatment using 50 % dimethyl formamide at room temperature following 7.1.9 (a part of reactive dye).	Treat the test specimen by boiling it in sodium hydroxide-hydrosulfite solution.	Using sodium sulfide, dye and ascertain the existence of sulfur, following 7.1.4 (sulfur dye or sulfur vat dye). Using hydrosulfite, dye following 7.1.5 (1) (vat dye or solubilized vat dye, other than indanthrene blue). Make the coloration test and ash test following 7.1.6 (oxidation dye). Make the pyridine boiling following 7.1.9 (some of reactive dye).
		If the test specimen cannot recolor	
		Make the chromium or copper test following 7.1.2 (1) [direct dye (metal aftertreatment or metalline)]. Make the pyridine boiling and the fluorescent test following 7.1.8 (a part of naphthol dye). Make the dimethyl formamide boiling following 7.1.2 (2) [direct dye(diazotization developing)] and following 7.1.9 (a part of reactive dye).	

7.1.2 Method of Class Identification for Direct Dye Put a test specimen into a test tube, and boil it in a solution having been obtained by adding 0.5 to 1.0 ml of concentrated aqueous ammonia to 5 to 10 ml of water to extract the dye perfectly.

Take out the specimen of which dye has been extracted, add 10 to 30 mg of undyed cotton cloth and 5 to 50 mg of sodium chloride to the extract liquid, boil the liquid for 40 to 80 sec and let it cool, and wash it with water. If the undyed cloth is coloured to the hue approximately similar to that of the specimen in this test, it can be determined that the dye used for the specimen is direct dye.

In addition, when extraction is done considerably at the extraction with 1 % aqueous ammonia after chloric acid treatment described in (1) below, if the undyed cloth is coloured to the hue approximately similar to that of the specimen, it can be determined that the dye used for the specimen is direct dye.

If the result of this test makes the identification difficult, identify the dye class following the procedure (1) or (2) below.

- (1) Method of Class Identification for Direct Dye (Metal Aftertreatment or Metalline) Boil the test specimen in 1 % hydrochloric acid solution of the volume 100 times as that of the specimen for 3 min, wash it well with water, put it into a test tube, and boil it in 5 to 10 ml of 1 % aqueous ammonia for 2 min. At this time, some of direct dye⁽³⁾ is extracted considerably and some of reactive dye is extracted, but some of direct dye of metal aftertreatment and diazotation developing and the others are extracted only a little or not at all.

If the extraction is done only a little or not at all, boil a fresh test specimen in 3 to 6 ml of water added with 1 to 2 ml of 10 % sodium hydroxide solution, further boil after adding 0.01 to 0.03 g of sodium sulfite for 2 to 5 sec, take out the specimen and wash with water, and put it on filter paper to expose to air. If this treatment with sodium hydrosulfite renders the specimen to change or to fade its colour and the washing with water thereafter causes no re-colouring⁽⁴⁾, make the test described in (a) or (b) below. In this test, if the existence of any one is ascertained, it can be determined that the dye used for the specimen is metal aftertreated or metalline direct dye.

Notes (3) This treatment makes a great part of common direct dye to be extracted considerably. Since this great part has already excluded as the result of the class identification specified in the text, the remaining dye is herein denoted in the term "some of".

- (4) Phthalocyanine (copper bearing) direct dye can recolour as the result of air oxidation.

Remark: Direct dye, including metal aftertreated or metalline and diazotation developing direct dye, is extracted rapidly, both when treated by boiling with N, N-dimethyl formamide as specified (2) below and when treated with 5 ml of 30 % pyridine solution at room temperature.

- (a) Chromium Confirming Test Put 0.1 to 0.2g of the test specimen in a porcelain crucible to ash it fully, and add 0.05 to 0.15 g of flux⁽⁵⁾ to fuse the specimen. If chromium exists, the fused matter presents orange yellow when it is hot and greenish yellow when it cools down.

Note (5) Mixture of same volume of sodium carbonate and of sodium nitrate.

- (b) Copper Confirming Test If the test specimen contains no chromium, ash the other specimen, solve the ash in approx. 0.25 to 0.50 ml of nitric acid, add 1 to 2 ml of water, and boil. Transfer the solution in a test tube, cool and add approx. 1 to 2 ml of aqueous ammonia (approx. 0.90 in specific gravity). If copper exists, the solution presents blue.

- (2) Method of Class Identification for Direct Dye (Diazotation Developing)
In the case where it is confirmed that, as the result of the test (1) above, the dye of the test specimen contains no metal and that, as the result of the test of 7.1.8, the dye is not naphthol dye, put the specimen in a test tube, add 5 ml of N, N-dimethyl formamide and boil. If the dye is extracted by this treatment, it can be determined that the dye used for the specimen is diazotation developing direct dye.

Remark: Put same volume of stearic acid and of uric acid in a small test tube with care, and heat to melt it. Into this solution, put a yarn of approx. 3 cm length picked out of the specimen. If diazotation developing direct dye exists, the lower layer is dyed more strongly than upper layer (stearic acid)⁽⁶⁾.

Note (6) Irrespective of existence or nonexistence and type of after-treatment, the lower layer is dyed on all occasions of direct dye.

In the case of basic dye and naphthol dye, the upper layer is dyed strongly, but in the case of reactive dye, both upper and lower layers are not dyed.

7.1.3 Method of Class Identification for Basic Dye If the extraction with aqueous ammonia specified in 7.1.2 is can be done only a little or not at all, proceed to the tests (1) and (2) below. If, in the test (1), the dye moves to acetic acid layer from ether layer to recolour to the hue same as that of the liquid extracted with glacial acetic acid, and further if, in the test (2), a tannin mordanted cotton cloth colours more deeply than a cotton cloth without mordanting, it can be determined that the dye used for the test specimen is basic dye.

- (1) Take a fresh test specimen in a test tube, add 0.25 to 0.5 ml of glacial acetic acid and heat, add 3 to 5 ml of water and boil, and then take out the specimen. Add 5 to 7 ml of 10 % sodium hydroxide solution to the extract liquid to make it alkaline, cool it down, add 3 to 5 ml of ethyl ether, and shake the test tube well while plugging the tube mouth with thumb. After leaving the test tube to stand still to allow the liquid to separate to two layers, take out the upper ether layer alone with a pipette, transfer the liquid to a fresh test tube, add 2 to 5 drops of 10 % acetic acid solution, and shake the tube thoroughly.